

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : B27K		A2	(11) International Publication Number: WO 97/12735 (43) International Publication Date: 10 April 1997 (10.04.97)
(21) International Application Number: PCT/NZ96/00107 (22) International Filing Date: 1 October 1996 (01.10.96)		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).	
(30) Priority Data: 280154 2 October 1995 (02.10.95) NZ		Published <i>Without international search report and to be republished upon receipt of that report.</i>	
(71) Applicant (for all designated States except US): NEW ZEALAND FOREST RESEARCH INSTITUTE LTD. [NZ/NZ]; Sala Street, Rotorua 3201 (NZ).			
(72) Inventors; and (75) Inventors/Applicants (for US only): NASHERI, Kourosh [NZ/NZ]; 6A Matipo Avenue, Rotorua 3201 (NZ). PENDLEBURY, James [GB/NZ]; 23 Exeter Place, Rotorua 3201 (NZ). DRYSDALE, Jeanette [NZ/NZ]; 265 James Fletcher Drive, Otahuhu 1006 (NZ).			
(74) Agents: ROGERS, Karl, Malcolm et al.; Greg West-Walker & Company, Todd Building, 8th floor, 171-177 Lambton Quay, Wellington 6001 (NZ).			
(54) Title: A METHOD FOR IMPREGNATION OF WOOD AND WOOD BASED PRODUCTS			
(57) Abstract <p>The invention relates to a method of impregnating wood or wood based products with a preservative or other wood modifying chemical under pressure. A composition comprising said chemical is impregnated into the wood under hydraulic pressure and the hydraulic pressure is controlled to control the amount of chemical impregnated into the wood. The hydraulic pressure is replaced with sufficient pneumatic pressure to hold the impregnated composition in the wood while the unused composition is removed from the treatment vessel. The pneumatic pressure remains applied to force the chemical further into the wood.</p>			

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AM	Armenia	GB	United Kingdom	MW	Malawi
AT	Austria	GE	Georgia	MX	Mexico
AU	Australia	GN	Guinea	NE	Niger
BB	Barbados	GR	Greece	NL	Netherlands
BE	Belgium	HU	Hungary	NO	Norway
BF	Burkina Faso	IE	Ireland	NZ	New Zealand
BG	Bulgaria	IT	Italy	PL	Poland
BJ	Benin	JP	Japan	PT	Portugal
BR	Brazil	KE	Kenya	RO	Romania
BY	Belarus	KG	Kyrgyzstan	RU	Russian Federation
CA	Canada	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	KZ	Kazakhstan	SG	Singapore
CH	Switzerland	LI	Liechtenstein	SI	Slovenia
CI	Côte d'Ivoire	LK	Sri Lanka	SK	Slovakia
CM	Cameroon	LR	Liberia	SN	Senegal
CN	China	LT	Lithuania	SZ	Swaziland
CS	Czechoslovakia	LU	Luxembourg	TD	Chad
CZ	Czech Republic	LV	Latvia	TG	Togo
DE	Germany	MC	Monaco	TJ	Tajikistan
DK	Denmark	MD	Republic of Moldova	TT	Trinidad and Tobago
EE	Estonia	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	UG	Uganda
FI	Finland	MN	Mongolia	US	United States of America
FR	France	MR	Mauritania	UZ	Uzbekistan
GA	Gabon			VN	Viet Nam

A METHOD FOR IMPREGNATION OF WOOD AND WOOD BASED PRODUCTS

TECHNICAL FIELD

5 This invention comprises a method of impregnation of lignocellulosic materials including wood and wood based products with preservatives and wood modifying agents such as fire retardants, water repellents, and/or wood densifying agents.

BACKGROUND ART

10

It is common to impregnate wood or wood based products with preservative chemicals. Commonly used wood preservatives include copper chrome arsenic (CCA), other copper chrome based or copper preservatives and other water-based inorganic preservatives based on boron for example. Wood is also commonly impregnated with other wood modifying agents such as fire retardants, water repellents, dimensional stabilising agents, and wood hardening agents for example. With the latter, impregnation may be followed by formation of insoluble polymers to add density and hardness to the wood. In many processes after impregnation with reactive preservatives or modifying agents it is desirable that the wood has excess impregnate chemical drained from it and the wood be subsequently heated to fix the chemicals in the wood.

Common commercially used methods of wood impregnation, with reactive wood preservative systems, for example chromium containing preservatives such as CCA, suffer from certain disadvantages.

First, following impregnation under hydraulic pressure, liquid comes out of the wood into the working chemical solution when the pressure is released. This "pressure kickback" is generally weaker and/or the ingredients are no longer in balance and it may also contain naturally occurring sugars and other molecules present in the wood which are carried back

5 out of the wood. When the hydraulic pressure is released, the kickback solution is normally returned directly into the working solution causing imbalances in the working solution chemical concentrations and ultimately sludging in the working solution and on the wood surface in subsequent charges. The rate of sludge build up is process dependent (solution turnover, volume etc). In addition, in many treatment schedules, a vacuum is finally

10 applied to remove excess liquid from the impregnated wood surface and chemically depleted solution will also come out of the wood during the application of the final vacuum (due to residual air in the wood moving to the wood surface). This solution is generally termed the "vacuum kickback".

15 Secondly, with existing methods where kickback occurs at the end of the process it is difficult to control the exact amount of the chemical impregnated into the wood. Fixation of chemicals such as CCA occurs naturally at ambient temperatures (usually above 7°C) during treatment of wood. However, with such fixation the treater has no control over the amount of deposition in the wood or the degree of fixation. Further, the treater has no

20 control over the disproportionation in the working solution. This difficulty means that where inadequate amounts, volumes or concentrates of chemicals are used, the core of the

wood is not treated with an adequate level of the chemical and where the amount of chemical is adequate to treat the core of the wood, the amounts of chemical in the outer section of the timber may be excessive, significantly adding to the cost of the treatment.

5 The existing methods generally show high variability in the amount of chemical deposited in the wood particularly for the frequently occurring situation in which the sample contains mixtures of sapwood and sometimes less permeable heartwood.

DISCLOSURE OF INVENTION

10

The invention provides an improved and/or at least alternative process which allows effective and controlled impregnation of wood or wood based products or other lignocellulosic material with wood preservative or other wood modifying chemicals without substantial depletion and/or contamination of the balance of the unimpregnated chemical.

15

In a preferred form the invention also allows a method of impregnating and fixing reactive wood preservatives.

20 In broad terms the invention comprises a method in which wood or wood based products or other lignocellulosic material impregnated with a wood preservative or other wood modifying chemical under pressure but the pressure impregnation phase of the method is separated into a hydraulic pressure phase and a pneumatic pressure phase. The hydraulic pressure phase impregnates the solution into the wood while the pneumatic pressure phase holds the impregnated chemical in the wood while the balance of the working chemical is

collected. By substituting the hydraulic pressure with pneumatic pressure whilst maintaining the same pressure or increasing the pressure, pressure kickback of chemical out of the wood and contamination of the balance of the working chemical is minimised or eliminated.

5

In the preferred method of the invention the hydraulic pressure is controlled so as to control the volume or amount of chemical impregnated into the wood and the pressure in the pneumatic pressure phase is increased above that used in the hydraulic pressure phase to force the chemical further into the wood.

10

In this preferred method the level of pneumatic pressure may be increased above that required to hold the impregnated composition in the wood, to force the chemical further into the wood, by immediately introducing into the treatment vessel, gas at a pressure level which will force the chemical further into the wood, or more preferably the pressure is 15 increased over time after initially introducing the pneumatic pressure at a lower level (eg a pressure equal to the hydraulic pressure) while the unimpregnated balance of the working chemical is removed.

After the collection of the unimpregnated balance of the chemical during the pneumatic 20 pressure phase, the pneumatic pressure is vented to atmospheric pressure and any resulting pressure kickback liquid from the wood may be collected and separated. A vacuum can then optionally be applied to extract any excess impregnate chemical or remove any residual liquid from the wood surface and any resulting vacuum kickback liquid may also be collected and separated.

Drainage of the unimpregnated treatment composition during the pneumatic pressure phase is carried out ideally at a pressure no lower than the pressure of the hydraulic pressure phase to avoid kickback of the treating composition.

5 The process of the invention thus separates the kickback solutions from a working solution during treatment and prevents water soluble sugars and extractives from contaminating the working solution, thereby eliminating the elemental depletion of the treating solution and risk of sludging or preservative solutions such as CCA. Preferably the unused solution is used in treatment of further batches of wood or wood products by the method of the

10 invention.

The process of the invention in at least preferred forms also allows for the absorption of impregnate chemical in a mixed charge of sapwood and heartwood to be manipulated, via adjustments to the level and time of the hydraulic and pneumatic pressure phases, to a pre-
15 determined level and thus improves the quality, and reduces the variability in chemical loading, of the treatment achieved over conventional processes. Preferably the pressures and times are chosen to allow chemical to be deposited in the core of the wood at a concentration of at least 75%, preferably at least 100%, of that in the outer one third of the cross-section of the wood.

20

The process of the invention allows a predetermined amount of chemical to be taken up by the wood in the hydraulic pressure phase. The volume of the wood and the volume of the treatment vessel can be determined by methods well known in the art so as to allow determination of the amount of working solution to be provided to give the required uptake

of treatment composition. Pneumatic pressure holds the predetermined amount of treatment composition in the wood while the unused treatment composition is removed from the vessel. In preferred embodiments of the invention, the pneumatic pressure is higher to deliver more of the treatment chemicals to the core of the wood. The same level of 5 delivery to the core of the wood may in existing processes be only achievable by using a greater uptake of working composition.

Control of the hydraulic pressure phase allows a high level of control of impregnation of the required volume of impregnation chemical into the wood. The resulting reduction of 10 coefficient of variation of the retention allows savings on chemical usage and better control over compliance with wood treatment standards.

The process of the invention in at least preferred forms reduces the net absorption of preservative and drippage from treated material post treatment when compared with 15 conventional schedules without loss in the quality of treatment achieved. This is of environmental significance for treatment plants who may move unfixed wet timber following treatment and must invest capital in protecting soil from any drippage.

The process of the invention also generally takes no longer to perform than any currently 20 available treatment schedule. Preferably the time the wood is under hydraulic pressure plus the time the wood is under pneumatic pressure is less than five hours (more preferably less than two or three hours in circumstances favourable for impregnation).

The process of the invention may be used to impregnate wood or wood based products or other lignocellulosic material. By wood is meant solid wood of all types whether sawn, dressed or otherwise, and by wood based products is meant all forms of reconstituted wood products such as particle board, fibre board including medium density fibre board, hard board, wafer board, bonded wood products such as finger jointed wood, laminated wood, plywood and similar.

The gas used in the pneumatic pressure phase may be air or nitrogen. Other gases may also be used.

10

Preferred pressures for the hydraulic pressure phase of the process range from 20-2000 kPa or more according to the permeability of the wood species, more preferably 400-2000 kPa, and most preferably 600-1500 kPa. Preferred pressures, more preferred pressures and most preferred pressures for the pneumatic pressure phase range from 20-2000 kPa, 400-2000 kPa and 600-1500 kPa respectively, provided that the pressure in the pneumatic pressure phase is ideally at least as high as during the hydraulic pressure phase. In a preferred form of the process the pressure during the pneumatic pressure phase is initially the same as during the hydraulic pressure phase and is subsequently increased to further drive the impregnate chemical into the wood.

15

Preferably after removal of the unused treating composition, the pneumatic pressure applied is at least 30% more than the pressure used during the hydraulic pressure phase. More preferably the increase is to at least 80% more than used in the hydraulic pressure phase.

For example an hydraulic pressure stage of +700 kPa may be followed after drainage at the same pressure and by a pneumatic pressure stage of +1400 kPa.

Typically the duration of the hydraulic pressure phase is 5-240 minutes, more preferably 5-180 minutes. The duration of the subsequent pneumatic pressure phase is preferably 5-240 minutes, more preferably 7-180 minutes. Factors affecting the pressure duration include the type of chemical being impregnated into the wood, the type of treatment required, the size of the piece or pieces of wood, the wood species (e.g. soft wood or hard wood), and the moisture content of the wood for example. Other important factors are the amount of penetration and retention of the impregnate chemical desired.

In a preferred form of the invention the hydraulic pressure used is in the range of 20-2000 kPa and after the unused composition is removed from the treatment vessel the pneumatic pressure used is 100-1000 kPa higher than used during the hydraulic pressure phase so as to force the chemical further into the wood. Preferably in this form of the invention the pneumatic pressure is at least 30% (more preferably at least 80%) higher than the pressure used in the hydraulic phase.

In another form of the invention the pneumatic pressure used to force the chemical further into the wood is

- (a) 30% higher than the pressure used in the hydraulic phase, and
- (b) in the range 26-2000 kPa.

More preferably the pneumatic pressure used is in the range 520-2000 kPa.

Most preferably the pneumatic pressure is in the range 780-1500 kPa.

In another preferred form of the invention the pneumatic pressure used to force the chemical further into the wood is

- 5 (a) at least 80% higher than the pressure used in the hydraulic phase and
- (b) in the range 36-2000 kPa.

In this form of the invention the preferred pneumatic pressure is in the range 720-2000 kPa, more preferably 1080-1500 kPa.

10

In preferred embodiments of the invention where the chemical used can be fixed in the wood, the higher pressures in the pneumatic pressure phase used to force the chemical further into the wood is maintained for a period sufficient to allow fixation of the chemical into the wood.

15

The term "a hydraulic pressure phase" includes submerging of the wood in liquid impregnate chemical which is in turn under pneumatic pressure to effectively apply hydraulic pressure to the wood.

- 20 Most preferably the impregnate chemical is heated prior to impregnation, or is impregnated under conditions which will heat the impregnate chemical and/or the wood to avoid the need for a separate heating step subsequent to impregnation to fix the treatment chemicals in the wood where this is accelerated using heat. Preheating of impregnate chemical means the wood takes up the hot solution and provides a more uniform heating of the wood to

allow consistent fixation throughout the wood. It is difficult to uniformly heat wood particularly when it is saturated as the large volume of water within the wood makes heat transfer throughout the wood difficult. Heat-induced fixation as in the process of the invention also avoids the need for a separate heating-fixation step and also the need to 5 transfer of impregnated timber to a separate heating vessel as is commonly done.

Heating may be up to 100°C in the case of aqueous solutions of treatment chemicals such as aqueous solutions of chromium containing treatment chemicals e.g. CCA, with 60-95°C being preferred. Most preferably the temperature at the wood surface is 60-75°C.

10

This preferred form of the process of the invention provides the benefits discussed above plus it provides a method of impregnating and fixing reactive water repellants, fire retardants, wood modifying agents or wood preservatives, for example containing copper and/or chromium such as CCA, into wood through the use of hot solutions to achieve 15 treatment and fixation in a one-step process.

This preferred form of the process of the invention can take less than four hours and as such is as fast, or faster, than many available processing options whether treatment followed by steam fixation or treatment followed by hot water fixation.

20

Another benefit of this form of the invention is that advantage can be taken of the greater solubility of chemicals such as disodium octaborate at higher temperatures, so as to provide a greater loading of chemical.

It is not essential to this preferred form of the process invention that the wood material be filleted and thus block-stacked wood material can in many cases be successfully processed. This preferred version of the process invention can produce touch dry material with little or no drippage or surface sludging. Less energy is required for any re-drying operations 5 when compared to either hot water or steam fixed material. In comparison with other available treatment options, little or no time is required, post-treatment, on a drip pad.

This preferred form of the process of the invention can fix chromium containing preservatives such as CCA in the wood without any or with only minimum 10 disproportionation of the active elements when compared to a conventional process. It also eliminates or at least reduces sludging in solutions, sludge deposition on surfaces or drippage of preservative containing kickback from wood post-treatment.

Unlike both steam and hot water fixation systems the preferred form of the process of the 15 invention fixes chromium containing preservatives such as CCA deep throughout the wood thus reducing the risk of environmental contamination from exposure to rain, or redistribution prior to fixation of the active ingredients under ambient conditions, or leaching by exposure of treated timber prior to completion of the fixation process.

20 In a preferred embodiment of the invention wood is impregnated with a chromium based wood preservative by a process comprising

- (a) heating the preservative solution and then impregnating said solution into the wood at a solution temperature in the range 60-95°C under hydraulic pressure;

(b) replacing the hydraulic pressure with sufficient pneumatic pressure using air or nitrogen to hold the impregnated preservative in the wood while the unused solution is removed from the treatment vessel to prevent kickback contamination of the unused solution; and

5 (c) maintaining the pneumatic pressure at a level at least as high as the pressure in the hydraulic pressure phase wherein the temperature at which the preservative is impregnated into the wood and the level and time of the hydraulic and pneumatic pressure phases is manipulated to control the amount of the preservative deposited in the wood.

10 Preferably the pneumatic pressure is increased to drive the preservative further into the wood after removal of the unused chemical.

A particularly preferred form of the invention is a method of impregnating wood with a chromium based wood preservative comprising:

15 (a) heating the preservative solution and impregnating said wood at a solution temperature in the range of 60-95°C under hydraulic pressure;

(b) allowing a predetermined amount of said solution to be impregnated into the wood;

(c) replacing the hydraulic pressure with sufficient pneumatic pressure, using air or nitrogen, to hold the impregnated preservative in the wood while the unused solution is removed from the treatment vessel to prevent kickback contamination of the unused solution;

20 (d) increasing the pneumatic pressure to force the preservative further into the wood; and

(e) maintaining the pneumatic pressure at a level higher than used in the hydraulic pressure phase for a time sufficient to allow fixation of most of the preservative within the wood.

5 In processes of the invention the hydraulic pressure phase and pneumatic pressure phase described may optionally be followed by a second hydraulic pressure phase and a second pneumatic pressure phase. Four such phases particularly in combination with higher pressure in the second hydraulic pressure phase relative to the first hydraulic phase, may be used to carry out successive treatments. The first hydraulic pressure phase impregnates

10 a first chemical into the wood and the first pneumatic pressure phase holds the chemical in the wood. A greater number of cycles may be used to impregnate three or more chemicals. The use of more than one cycle may be useful where wood is being treated with two reactants, or a reactant and a catalyst for example. In other circumstances it may be useful where wood is to be treated with one chemical such as a wood preservative

15 throughout wood, and a second chemical such as a fire retardant primarily closer to the surface of the wood.

In some preferred forms of the invention hot impregnate chemical is held in contact with the wood prior to application of the hydraulic pressure phase. This heats the wood and

20 causes air in the wood to expand and leave the wood. When the hydraulic pressure is applied the impregnation of the chemicals into the wood is more rapid and any need to pull a vacuum initially is eliminated. Preferably the wood is left in contact with the chemical for 5-20 minutes before application of the hydraulic pressure.

The process of the invention may be used to impregnate wood preservatives such as copper chrome arsenic, copper chrome boron, copper azole, copper chrome zinc fluoride, other copper chrome containing formulations and boron preservatives. Chrome based wood preservatives are particularly preferred for use in the invention, both as salts and as oxides.

5 The process of the invention may also be used to impregnate fire retardants, water repellants, wood densifying agents, dimensional stabilising agents, isocyanates, wood hardening agents and similar. The impregnation conditions used will vary according to the nature of the chemical. Thus while hot treatment is preferred with chromium based preservatives, treatment with isocyanates is usually performed at ambient temperatures for

10 example.

Fire retardants such as borate salts (eg disodium octaborate) are preferred chemicals for use in the invention.

15 A further form of the invention provides wood or wood products treated with a preservative or other wood modifying chemical by a process of the invention.

BRIEF DESCRIPTION OF FIGURES

20 The invention will now be described by way of example only with reference to the accompanying figures wherein:

Figure 1 is a schematic diagram of one arrangement of treatment plant for carrying out the process of the invention, and

Figure 2 shows pressures and temperatures during the course of a treatment of wood in treatment W of Example 5.

DESCRIPTION OF PREFERRED TREATMENT PLANT

5

The treatment plant of Figure 1 comprises treatment cylinders 1, 2 and 3 and a working solution tank 4 containing treating solution 5. Working solution tank 4 has inlets 6 and 7 for receiving water and preservative concentrate respectively and an outlet 8 linked to a pipeline 9 which supplies treating solution 5 to treatment cylinders 1, 2 and/or 3 when appropriate according to the settings of valves in the pipeline. Hydrostatic pressure is applied using pump 10. A second pipeline 11 allows provision of compressed air to cylinders 1, 2 and 3 via inlets 12 when the appropriate valves are open. Each treatment cylinder 1, 2 and 3 is provided with a vacuum line 13 containing a condenser 14. Each cylinder 1, 2 and 3 also has an outlet 15 for kickback and an inlet/outlet 16 for receiving treating solution from pipeline 9 and for drainage of unused treatment solution at the end of the hydraulic phase. Figure 1 shows timber 17 being impregnated with treating solution 5 in cylinder 1. In cylinder 2 treating solution is being held within and forced further within timber by compressed air in the pneumatic phase. Meanwhile in cylinder 3 the pneumatic pressure has been released and the cylinder door 18 has been opened to allow removal of the timber.

The treating solution 5 is heated and filtered in a process line 19 running out of and back into the working solution tank 4 and having a heat exchanger 20 which receives steam on the heating side 21. This process line also has a pump 22 and a 200 micron filter 23.

Such a treatment plant allows different phases of the process of the invention to be carried out at the same time.

One plant may comprise several impregnation cylinders as shown in Figure 1. Such an 5 integrated plant would have an increased throughput capacity, reduced chemical storage holding and reduced energy usage when compared with a conventional treatment plant. The working solution is used efficiently (which is especially important for hot solutions) as the time the solution is not in use is reduced.

10 The main reason why the process of the invention lends itself to this throughput improvement lies in the control of the pneumatic pressure phase to hold the required amount of liquid in wood for further depth of penetration and degree of fixation.

Conventional processes perform the same operation, but only use hydraulic pressure and 15 as such tie up the working solution during this phase rendering it unavailable for other plants. Thus if the new process is employed it is possible to have a mother working solution tank in the middle of 3, 4 or more satellite treatment (ie impregnation) plants.

EXAMPLES

20

The invention is further illustrated by the following examples:

EXAMPLE 1**MATERIALS AND METHODS**

Four 5 metre long 100x50 mm *Pinus radiata* sapwood and four 5 metre long 100x50 mm 5 *Pinus radiata* heartwood boards were selected from the green chain of Waipa sawmill. All the boards were then high temperature dried to 8% MC using a commercial 120°/70°C schedule.

The treatment plant utilised in the trials consisted of two, interconnected, vertically 10 orientated, stainless steel vessels. Each vessel was 1.2 metres long and had an internal diameter of 150 mm. Each vessel was capable of being independently evacuated, or pressurised. During the trials, vacuums were drawn using a conventional vacuum pump. However all the hydraulic pressures were obtained through the use of compressed air and nitrogen pressure on liquid surfaces rather than by conventional means.

15

After drying each board was dressed to 95x45 mm and then cross cut into fourteen end matched 294 mm long samples per board. These samples were then randomly allocated (one per process) to each of the nine treatment processes under evaluation. Each sample was then end sealed with two coats of aluminium sealer to stop end penetration of 20 preservative during treatment and weighed. A total of eight samples (four sapwood and four heartwood) were used per treatment process.

TREATMENT PROCESSES

In all cases the treatment plant, wood and treating solution were maintained at ambient temperature (between 19° and 22°C). All treatments were performed using Koppers Hickson supplied CCA type C preservative.

5

All treatments followed the following format, though for treatments 8 and 9 there was no initial vacuum and flooding was at atmospheric pressures.

Initial Vacuum

10 Flood under Vacuum

Hydraulic pressure

Drain under a pressure (collect working solution) and continue pressurising (using compressed nitrogen)

Release pressure (collect pressure kickback)

15 Final vacuum (collect vacuum kickback)

Times, pressures and the results are summarised in Table 1.

In all nine treatment schedule evaluations the *working solution* (considered as the 20 preservative solution which was in contact with wood during the hydraulic pressure phase) was drained under constant pressure by admitting compressed air to replace the solution drained. The volume of working solution drained in each treatment was measured and samples were collected for analyses and sludge formation determination.

The pressure kickback solution was considered as that solution obtained during and immediately after the gradual release of a schedule's "air pressure" period. The volume of pressure kickback solution obtained was measured and samples were collected for analyses and sludge formation determination.

5

After collection of all the pressure kickback solution a final vacuum period of 15 minutes was applied in each schedule. This final vacuum period extracted solution from the wood and this was considered as *vacuum kickback solution*. The volume of vacuum kickback solution obtained was measured and samples were collected for analyses and sludge 10 formation determination.

The air dried samples were then cross cut into a series of sections for core cross section analysis and spot testing. The central cross sectional surface of each sample was sprayed with 5% ammonia solution followed by 0.5% rubeanic acid and this resulted in any copper 15 containing areas turning almost black after drying. In addition six samples from each treatment (3 sapwood and 3 heartwood) were selected for further analysis of preservative distribution. The sections were then analysed for copper, chromium and arsenic in accordance with AWPA A11-83.

20 RESULTS

The sapwood and heartwood uptakes and amount of solution drippage for each treatment are summarised in Table 1.

TABLE 1 Summary of 9 treatments

Treatment	Process Schedule	Uptake l/m ³				Sap/Heart Uptake Ratio %
		Total samples	Sapwood samples	Heartwood samples	Heart: after drainage	
1	kPa Minutes	Mean 3	Mean 4	Mean 4	Mean 4	61
	-35 10	samples	samples	samples	samples	
	1400 Hydraulic 5	324.7	245.7	403.6	..	
	1400 Pneumatic 120	CV %	CV %	CV %		
2	kPa Minutes	Mean 3	Mean 4	Mean 4	Mean 4	51
	-35 10	samples	samples	samples	samples	
	1400 Hydraulic 15	347.8	233.8	461.9	..	
	1400 Pneumatic 120	CV %	CV %	CV %	..	
3	kPa Minutes	Mean 3	Mean 4	Mean 4	Mean 4	42
	-35 10	samples	samples	samples	samples	
	1400 Hydraulic 60	412.8	243.6	582	..	
	1400 Pneumatic 60	CV %	CV %	CV %		
4	kPa Minutes	Mean 3	Mean 4	Mean 4	Mean 4	44
	-35 10	samples	samples	samples	samples	
	1400 Hydraulic 120	431.6	263.1	600.1	509.1	
	1400 Pneumatic 0	CV %	CV %	CV %		
5*	kPa Minutes	Mean 3	Mean 4	Mean 4	Mean 4	41
	-35 10	samples	samples	samples	samples	
	1400 Hydraulic 20	335.5	196.5	474.2	445.0	
	1400 Pneumatic 0	CV %	CV %	CV %		
	-90 15	42	4.2	3.6		
	-90 45					

* Dry process (Commercial modified Bethell process)

** Not recorded

TABLE 1 (continued)

Treatment	Process	Total	Uptake l/m ³				l/m ³ dripped from Heartwood	Sap/Heart Uptake Ratio %
			Sapwood	Heartwood after drippage	Heart	Mean 4 samples		
6	kPa Minutes	Mean 8	Mean 4	Mean 4	Mean 4			
	-35 15	samples	samples	samples	samples			
	700 Hydraulic 10	234.0	246.5	222.3	162	60.3	110	
	1400 Hydraulic 120	CV %	CV %	CV %				
7	kPa Minutes							
	-35 15	275.2	268.8	277.6				
	700 Hydraulic 20	CV %	CV %	CV %	189.5	85.2	97	
	1400 Hydraulic 120	10.7	9.3	11.3				
8	kPa Minutes							
	0 0	276.7	235.2	319.2		97.8		
	700 Hydraulic 20	CV %	CV %	CV %	220.5	Some sapwood dripped	74	
	1400 Hydraulic 120	19	13.3	11.3				
9	kPa Minutes							
	0 0	307.4	190.9	424				
	1400 Hydraulic 10	CV %	CV %	CV %	276.9	147.1	45	
	1400 Hydraulic 120	38	4.7	2.3				
	.90 15							

From Table 1 it is quite obvious that by adopting different process schedules the heartwood absorption can be controlled. Particularly uniform sapwood/heartwood preservative uptake was produced by treatments 6 and 7.

- 5 Analysis of preservative penetration via spot testing, for copper penetration in sample cross sections indicated that uniform and complete sapwood penetrations were achieved using all nine treatment schedules. In contrast heartwood penetrations were process dependent. In general, processes with higher heartwood uptakes had more uniform heartwood penetrations.
- 10 Sludge formation in treating, working, pressure kickback and vacuum kickback solutions were determined by storing solution samples in cylindrical sample jars in the laboratory at a temperature between 18°C to 24°C and observing the formation of sludge. The sludge formations observed for all nine treatment processes evaluated can be summarised as follows:
 - 15 Solution before treatment did not show any sludge after three months;
 - Working solution did not show any sludge after three months;
 - Pressure kickback solutions showed some sludge formation in less than 16 hours after treatment;
 - 20 Vacuum kickback solutions showed some sludge formation in less than 16 hours after treatment;

All these solutions were analysed later for Cu, Cr and As and the analyses demonstrated that during treatment some fixation of elements had occurred. The amount of elemental fixation that occurred during treatment is dependent on the solution temperature and treatment process variables like total treatment time, the amount of solution in wood during this period, pressure and vacuum duration.

The analytical retention of all treatments are given in Table 2:

Table 2

10	Treatment	Mean 6 Sample Retention TAE (% w/w)	6 Sample Retention (%cv)	Mean 3 Sapwood Retention TAE (% w/w)	Mean 3 Heartwood Retention TAE (% w/w)
1	0.781	18.1	0.858	0.704	
2	0.847	9.4	0.817	0.877	
3	0.991	18.4	0.864	1.118	
4	0.947	13.6	0.835	1.059	
15	5	0.774	23.4	0.634	0.914
6	0.645	30.1	0.825	0.465	
7	0.701	22.0	0.832	0.570	
8	0.732	16.7	0.773	0.690	
20	9	0.790	13.4	0.728	0.853

20

This example illustrates that another improvement by using a pneumatic pressure step is the production of more uniform treated end product. Table 2 shows by using Treatment 2 at an ambient temperature a significant improvement of 14% reduction in coefficient of variability achieved in the chemical retention when compared with the same matched timber pieces treated by conventional modified Bethell process (Dry process treatment 5).

This improvement greatly reduces the chemical usage. (Treatment 2 mean 6 samples retention 0.847% w/w and Cv% 9.39, Treatment 5 mean 6 samples retention 0.774% w/w and Cv% 23.42).

5 The analysis of the wood samples from each of the nine treatment schedules evaluated demonstrated that a uniform penetration of preservative occurred throughout the cross section of all of the sample analysed. The analytical core/cross section retention ratios, given in Table 3, are a good indication of this uniformity.

10

Table 3: Percentage core/cross section retention ratio

15

Treatment	Mean 3 Sapwood Core/Cross Ratio	Mean 3 Heartwood Core/Cross Ratio	Average Mean 6 Core/Cross Ratio
1	92.5	116.8	104.6
2	95.5	101.7	98.6
3	89.8	106.3	98
4	90.3	113.7	102
5	90.3	108.5	99.4
6	94.2	121.0	107.5
7	95.3	87.2	91.2
8	95.6	103.0	99.3
9	91.8	110.0	100.8

20

EXAMPLE 2

MATERIALS AND METHODS

25 The materials, methods and treatment plant are as described in Example 1.

A total of eight samples (four sapwood and four heartwood) were used per treatment process.

A combination of hot water and an immersion heater in the working solution tank were 5 used to make the CCA solution up to the required solution temperature in treatments 10 and 11. In treatment 12 hot water was used to make up the initial solution and this was then further heated by bubbling high pressure dry steam through the hot solution in the working tank.

10 TREATMENT PROCESSES

The three treatment processes evaluated using hot CCA solutions were as follows. In all cases the temperatures in the treatment plant and treating solution were monitored and recorded through the use of thermocouples and a data logger. All treatments were performed using Koppers-Hickson supplied CCA type C oxide preservative.

15

Treatment 10

No initial vacuum

Solution temperature before flooding 89°C

Flood under atmospheric pressure

20 Solution temperature after flooding 78°C

Hydraulic pressure 700 kPa for 20 minutes

Drain at 700 kPa pressure (collect working solution) increase the pressure to 1400kPa and continue pressurising (using compressed nitrogen) for 120 minutes.

Release pressure @ 200 kPa per minute (collect pressure kickback)

Final vacuum of -80 kPa for 15 minutes (collect vacuum kickback)

Treatment 11

As for treatment 10 except that solution temperatures before flooding and after flooding
5 were 96°C and 75°C respectively and the time for the hydraulic pressure phase was 7
minutes and the pressuring with compressed nitrogen continued for 133 minutes.

Treatment 12

As for treatment 10 except that no initial vacuum solution temperatures before and flooding
10 after flooding were 92°C and 88°C respectively and the hydraulic pressure used was at 500
kPa. The working solution was continuously heated.

Working solution, pressure kickback solution and vacuum kickback solution were collected
separately as in Example 1. After treatment the air dried wood samples were analysed in
15 accordance with AWPA A11-83 as for Example 1.

Results

The sapwood and heartwood uptakes and amount of solution drippage for each treatment
are summarised in Table 4.

TABLE 4: Summary of Hot solution treatments

Treatment	Process	Uptake l/m ³				l/m ³ dripped from Heartwood	Sap/Heart Uptake Ratio %
		Total	Sapwood	Heartwood	Heart after drip		
10	kPa Minutes	Mean 8 samples	Mean 4 samples	Mean 4 samples	Mean 4 samples		
	0 0	316.4	184.2	448.6	399	49.5	41.1
	700 Hydraulic 20	CV %	CV %	CV %			
	1400 Air 120	42	9.7	5.6			
11	kPa Minutes						
	0 0	239.2	183.1	295.3	248.1		
	700 Hydraulic 7	CV %	CV %	CV %		47.2	62
	1400 Air 133	27	12	13			
12	kPa Minutes						
	0 0	247.9	202.0	293.7	264.3		
	500 Hydraulic 20	CV %	CV %	CV %		29.4	84.3
	1400 Air 120	25	21.8	14			
	-80 15						

Treatment schedule 10 utilised exactly the same process parameters as the treatment schedule 8 reported in Example 1 with one important exception, namely the preservative solution temperature. Schedule 10 used a solution temperature of between 78 and 89°C whereas schedule 8 used a solution temperature of 20°C.

Given that the treatment cycles for the two schedules were identical, examination of their respective results indicates that the use of hot solutions reduces the volume of heartwood drippage post treatment. This reduction occurs because the hot solution cools down inside the heartwood after treatment and contracts, forcing the remaining air trapped in the wood also to contract and thus hold the solution in the wood.

Analysis of the preservative penetration by spot testing the sample cross sections for copper penetration indicated that uniform and complete sapwood penetrations were achieved using all three treatment schedules evaluated. In contrast the heartwood penetrations were process dependent such that processes with the higher heartwood uptakes had the more uniform heartwood penetrations.

Sludge formation in treating, working, pressure kickback and vacuum kickback solutions was determined as in Example 1. The degree of sludge formation observed for all solutions and treatments can be summarised as follows:

5 * Solutions before treatment did not show any sludge after one month
* Working solutions did not show any sludge after one month

Pressure kickback solutions for treatments 10, 11 and 12 which used hot solution did not show any sludge after one month. These solutions were clear.

10

Vacuum kickback solutions for treatments 10, 11 and 12 which used hot solution did not show any sludge after one month. These solutions were clear.

Preservative solution was collected from each stage of treatment processes 10, 11 and 12.

15 All these solutions were analysed for Cu, Cr and As and the results, given in Table 5, demonstrated conclusively that fixation of preservative elements occurs during treatment. By using hot solution in treatments 10, 11 and 12, almost all of the CCA solution fixed into the wood.

TABLE 5: Results of elemental analysis of treatment solutions

SOLUTION	ELEMENTS (g/100 mL)	Treatment 10	Treatment 11	Treatment 12
Solution Before Treatment	Cu	0.203	0.171	0.186
	Cr	0.344	0.307	0.325
	As	0.233	0.221	0.237
Working Solution	Cu	0.206	0.168	0.170
	Cr	0.343	0.300	0.309
	As	0.237	0.217	0.227
Pressure Kickback	Cu	0.00816	0.00630	0.00339
	Cr	0.02250	0.02030	0.00124
	As	0.00566	0.00540	0.00135
Vacuum Kickback	Cu	0.00388	0.00280	0.00352
	Cr	0.01510	0.01310	0.00157
	As	0.00169	0.00160	0.00217

EXAMPLE 3

Another 5 matched sets of radiata pine were treated as in Table 6:

TABLE 6 TREATMENT SCHEDULE

5

Treatment	A	B	C	D	E	F
CCA Temperature C°	12	95	90	70	90	95
Vacuum Before (kPa)	40	40	40	80	Soak	Soak
Duration (minute)	10	10	10	15	10	10
Pressure Dur (kPa)	215	215	215	215	215	215
Duration (minute)	60+90	60+90	60+90	60+90	60+90	60+90
Vacuum After (kPa)	90	90	86	86	86	90
Duration (minute)	15	15	15	15	15	15
Heating during treatment	No	No	Yes	Yes	Yes	No
Wood surface After	Clear	Clear	Sludge	Sludge	Sludge	Clear

Key to table : Before = Before treatment Dur = During treatment

After = After treatment 60+90=60 minutes hydraulic pressure +90 minutes air pressure

20 Flooding the very hot solution of CCA in treatments B and C caused a fast drop in temperature and vacuum. Thus in treatment D the initial solution temperature dropped to 70° C to facilitate the higher initial vacuum of 80 kPa. In treatments E and F the hot CCA was maintained in contact with the wood for 10 minutes in order to provide the initial vacuum (air expansion in the wood due to heating creates vacuum). During this time the wood became heated as well.

25

Heating the CCA before treatment (B and F) did not lead to sludging whereas heating during treatment (C, D and E) was associated with sludging.

EXAMPLE 4

5 In-situ fixation of CCA into wood is shown in the examples 1 to 3. This example illustrates the potential of the invention on alternative chemicals:

- A Fire Retardant (Disodium Octaborate Tetrahydrate)
- Copper Chrome Boron (CCB)
- 10 • Tanalith E (Copper Azole)

Solution Preparation

A fire retardant treating solution was made with Disodium Octaborate Tetrahydrate powder.

A 22 litre working solution of 48.1 g/100mL BAE (Boric Acid Equivalent) concentration
15 was prepared.

The CCB solution was made using Kamesan's formulation as follows:

Copper Sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$)	35.3%
Sodium Dichromate ($\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$)	47.1 %
20 Boric Acid (H_3BO_3)	17.6 %

A target solution of 2% was made using $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and analytical grade $\text{Na}_2\text{Cr}_2\text{O}_7$ and H_3BO_3 .

Tanalith E is a commercially-available wood preservative containing the active ingredients of copper, 12.4%(w/v), Boric acid, 5.6%(w/v), and Tebuconazole, 0.5%(w/v).

The Tanalith E treating solution was made by slowly adding concentrate Tanalith E into 5 hot water then heating to the required temperature. The solution was made to a target concentration level of 0.5 g/100mL of Copper, which equates to a 4.1 g/100mL of Tanalith E solution.

The treating solutions were prepared by dissolving the above required chemicals into hot 10 water and diluting to the required volume. They were then heated to a target temperature of 85°C with a submersible electric element.

Treatment Process.

An outline of the treatment process is as follows:

- 15 No initial vacuum
 Solution temperature before flooding 85°C
 Flood under atmospheric pressure
 Solution temperature after flooding 70°C
 Hydraulic pressure 700 kPa for 20 minutes
- 20 Drain at 700 kPa pressure (collect working solution) maintain the same pressure (using compressed air), 60 minutes for fire retardancy and 150 minutes for CCB and Tanalith E
 Release pressure @ 200 kPa per minute (collect pressure kickback)
 Final vacuum of -85 kPa for 20 minutes (collect vacuum kickback)

The two separate kickbacks were combined and an overall kickback sample collected and analysed.

Results:

5 i. Fire Retardency.

For this treatment eight samples were treated, 4 sapwood and 4 heartwood.

As was expected the Boron did not fix when in contact with the wood during the process.

10 This is shown by the fact that the kickback has a BAE approximately the same as the starting solution, 47.4 g/100 mL compared to 48.1 g/100 mL BAE respectively.

A benefit was in increasing the solubility of Disodium Octaborate Tetrahydrate powder to 48.1% by heating the treating solution to 85°C. Thus nearly half of the weight gain of the 15 wood during treatment is of fire retardency salt. With the invention the required uptakes are still achieved but are reduced to an average of 285 L/m³, therefore 135 kg/m³ of fire retardant salt is absorbed by the wood while the remaining 150 L/m³ was water. The wood was touch dry and drip free after treatment.

20 ii. CCB

The pneumatic phase was extended in the CCB treatment to 2.5 hours.

For this trial 10 samples were used, 5 sapwood and 5 heartwood.

The kickback had reduced colour compared to the solution before treatment. However the analytical results show that the fixation of the chromium was not as high as found with 5 CCA solutions. The level of chromium in the kickback was 0.092 g/100mL, which is 31% of the chromium in the solution before treatment. The net uptake of the wood during treatment was 300 L/m³. The results indicate that higher temperatures and/or longer fixation time may be preferable for CCB.

10 iii. Tanalith E

For the Tanalith E treatment 5 sapwood and 5 heartwood samples were treated.

The fixation of the copper was 67% of the original copper solution concentration. The 15 fixation of the H₃BO₃ and Tebuconazole was 40% and 88% respectively as found by differences between kickback and initial solution strength concentrations. The results indicate that higher temperatures and/or longer fixation time may be preferable for Copper Azole.

20 EXAMPLE 5

Twenty four trials using the invention were performed on a pilot-scale plant. The quality of the treatments achieved was evaluated by assessing the working solution after repetitive trials using air-dried, kiln-dried and pre-steamed wood. Preservative absorption and retention from Hazard Classes H3 to H6 (New Zealand Timber Preservation Council

standard) and resin bleeding on the surface of the wood was also investigated. The strength and balance of the Copper Chrome Arsenic Oxide (CCA oxide) working and kickback solutions produced by the processes was also analysed.

- 5 Twenty four semi-commercial trials were performed on a pilot-scale plant under the following conditions:

Treatment plant

The treatment plant used consists of a 2.4m long 1300 litre volume pressure cylinder with

- 10 a 1500 litre chemical work tank

The work tank solution was heated and filtered in a process line running out of the bottom of the solution tank and into the top. Heating was achieved by a 16 kW heat exchanger which received 130°C steam on the heating side. Flow was regulated by a 1.5kW variable

- 15 speed mono pump with a 200 micron 2" filter position immediately prior to the pump.

Solution temperature before all treatments was adjusted to 75°C.

Timber

- 20 A variety of timber sizes and types were used. All timber samples were 2.4 m long but varied in width and breadth. Several timber sample parameters were varied and included a mixture of sapwood / heartwood, kiln-dried, air-dried and pre-steamed radiata pine. Samples were treated to New Zealand Hazard Classes H3 to H6 (ref: NZS MP 3640).

Timber sizes and their moisture contents before and after treatment are summarised in Table 7. In all cases the charges were treated in block stack with strapping.

Table 7: Summary of all timber treated in example 5

AD=air-drying KD=kiln-drying

Treatment I.D.	No. of Samples Measured	Timber Cut	Size (mm)	Drying Schedule	Hazard Class
A	10	—	200 * 50	AD	H3
B	10	—	100 * 50	AD	H3
C	10	—	150 * 50	AD	H3
D	10	—	200 * 50	AD	H4
E	10	—	100 * 50	AD	H4
F	10	—	150 * 50	AD	H4
G	10	—	200 * 50	KD	H3
H	10	—	100 * 50	KD	H3
I	10	—	150 * 50	KD	H3
J	10	—	200 * 50	KD	H4
K	10	—	100 * 50	KD	H4
L	10	—	150 * 50	KD	H4
M	10	—	90 * 90	Steamed	H4
N	9	4 * 5 ↗	180 ↗	Steamed	H4
O	10	3 * 7 ↗	178 ↗	Steamed	H5
P	10	2 * 8 ↗	181 ↗	Steamed	H5
Q	3	—	228 ↗	Steamed	H5
R	3	—	241 ↗	Steamed	H6
S	14	—	125 * 125	Steamed	H5
T	14	—	125 * 125	Steamed	H5
U	15	—	100 * 100	Steamed	H5
V	15	—	100 * 100	Steamed	H5
W	19	—	100 * 100	Steamed	H5
X	18	—	100 * 100	Steamed	H5

CCA and Solution Heating

All treatments were performed using Tanalith CCA Type C Oxide preservative. For each charge approximately 1300 litres of pre-mixed CCA solution was heated to the required temperature of 75°C. All trials were preformed as soon as the working 5 solution reached 75°C.

Treatments

After flooding, the hydraulic impregnation phase was achieved by regulating air pressure onto the surface of the working solution. This pressure was transmitted through to the 10 treatment cylinder as hydraulic pressure. The hydraulic pressures used ranged for the 24 samples ranged from 619.6 kPa to 758.7 kPa.

At the end of the hydraulic pressure phase the treatment solution was drained from the treatment cylinder (at the same or higher air pressure) by slowly releasing the air 15 pressure in the work tank whilst admitting air pressure directly into the treatment cylinder. Once the working solution had drained from the treatment cylinder the air pressure in the treatment cylinder was further increased and maintained for a set time period. This constituted the pneumatic phase of the process. The increased pressures maintained were in ranges from 947-1117 kPa.

At the end of pneumatic phase and before the total pressure was released the residual working solution in the treatment cylinder (ie. that which resulted from: drippage from the wood, or bogey, or pipework contamination) was collected. The pneumatic pressure was then released in a controlled manner and the resultant volume of pressure kickback 5 was measured.

A final vacuum was drawn on the treatment cylinder range -81 kPag to -91 kPag for a pre-determined period. This vacuum was further improved by using a condenser to condense vapours generated from hot wood under vacuum. Upon release of the vacuum 10 to air the vacuum kickback was collected prior to removal of the charge. At the end of treatment vacuum condensate was also collected.

Treatment processes

Treatment time for all phases of 24 treatments A to X are summarised in Table 8.

Table 8: Process time for each treatment

Treatment	Flooding	Hydraulic	Drainage	Pneumatic	Vacuum	Total Process
I.D.	Period	Period	Period	Period	Period	Time
	Minutes	Minutes	Minutes	Minutes	Minutes	Minutes
A	7.0	22.0	9.0	125.0	12.0	175
B	5.0	30.0	11.0	119.0	17.0	182
C	6.0	26.0	9.0	123.5	20.3	185
D	5.0	40.0	9.0	130.0	20.0	204
E	4.0	40.3	6.0	132.0	18.0	200
F	5.0	40.0	8.0	124.7	19.2	193
G	4.0	26.3	9.0	128.3	22.2	190
H	5.0	26.0	10.0	133.0	18.3	190
I	5.5	20.0	8.0	127.2	16.2	177
J	2.8	40.7	4.0	129.0	19.0	193
K	3.0	43.0	6.0	124.0	16.0	192
L	3.7	40.0	9.0	127.5	19.2	199
M	2.8	40.3	7.0	128.5	19.5	198
N	3.0	61.5	6.0	130.0	20.0	221
O	5.0	42.0	7.0	126.5	30.0	211
P	5.0	42.0	6.0	131.3	30.5	213
Q	3.6	61.8	7.0	131.7	31.0	234
R	4.1	61.4	7.4	130.5	41.0	246
S	3.9	40.8	5.0	129.0	40.8	219
T	3.8	61.0	5.0	130.0	40.0	239
U	3.0	1.6	4.9	183.5	41.3	234
V	4.9	0.5	8.7	130.2	41.0	185
W	4.3	11.8	5.5	131.0	41.2	194
X	4.3	4.9	6.1	131.5	39.9	187

30

In treatment W (refer to Table 8) after the ramping up of pneumatic air pressure the treatment plant was sealed and the compressor disconnected. Figure 2 shows that during this period which lasted for 2 hours, the air pressure did not drop in the 1300 litre treatment plant. Steam and hydraulic pressure systems in a large plant can 35 experience relatively high reductions in pressure if the pressure source is disconnected.

The timber produced from treatments W was touch dry, drip free with no extra kickback contamination.

Preservative solution analyses

5 Solution analyses was carried out for solutions before treatment, working and kickback solutions. Solution analyses covered analysis of Cu, Cr, As and Cr^(VI). The kickback solution consisted of pressure kickback, vacuum kickback and vacuum condensate. The results of the above analyses are shown in Table 9.

Table 9: Solution analyses for all treatments

Treatment No.	Solution Before Treatment				Working Solution				Kieldback Solutions			
	Cu g/100ml	Cr g/100ml	As g/100ml	TAE	Cu g/100ml	Cr g/100ml	As g/100ml	TAE	Cu g/100ml	Cr g/100ml	As g/100ml	TAE
A	0.078	0.132	0.094	0.304	0.077	0.130	0.088	0.295	0.0189	0.01660	0.00630	0.02479
B	0.116	0.206	0.142	0.464	0.114	0.202	0.136	0.452	0.00258	0.01770	0.00272	0.02300
C	0.118	0.205	0.137	0.460	0.119	0.206	0.134	0.459	0.00344	0.01140	0.00332	0.01716
D	0.207	0.375	0.241	0.823	0.205	0.366	0.232	0.803	0.00317	0.01970	0.00149	0.02436
E	0.209	0.365	0.241	0.815	0.207	0.359	0.236	0.802	0.00283	0.01640	0.00136	0.02059
F	0.214	0.372	0.248	0.834	0.217	0.376	0.246	0.839	0.00374	0.02420	0.01730	0.04524
G	0.108	0.202	0.137	0.447	0.109	0.201	0.132	0.442	0.00255	0.00278	0.00203	0.00736
H	0.103	0.184	0.117	0.406	0.106	0.185	0.117	0.408	0.00215	0.00239	0.00111	0.00365
I	0.100	0.1174	0.123	0.397	0.101	0.171	0.123	0.395	0.00226	0.00601	0.00211	0.01038
J	0.219	0.377	0.247	0.843	0.211	0.363	0.235	0.809	0.00323	0.00403	0.00241	0.00967
K	0.208	0.368	0.234	0.810	0.213	0.371	0.232	0.816	0.00265	0.00585	0.00134	0.00984
L	0.217	0.379	0.235	0.831	0.217	0.373	0.250	0.840	0.00272	0.00840	0.00180	0.01292
M	0.273	0.463	0.312	1.048	0.272	0.457	0.304	1.033	0.00305	0.00880	0.00374	0.01739
N	0.360	0.664	0.420	1.444	0.360	0.655	0.407	1.422	0.00891	0.02355	0.00607	0.03853
O	0.315	0.975	0.573	2.063	0.514	0.964	0.568	2.046	0.01248	0.03608	0.00848	0.05704
P	0.343	0.980	0.615	2.138	0.554	0.960	0.598	2.112	0.01174	0.0375	0.00735	0.05284
Q	0.321	0.943	0.638	2.102	0.524	0.952	0.661	2.137	0.01920	0.04600	0.00640	0.01760
R	0.905	1.390	1.120	3.615	0.920	1.620	1.150	3.690	0.04560	0.1900	0.02000	0.18460
S	0.745	1.300	0.906	2.951	0.748	1.280	0.909	2.937	0.02190	0.06500	0.01160	0.03850
T	0.416	0.715	0.490	1.641	0.412	0.730	0.473	1.615	0.01190	0.04080	0.00820	0.06090
U	0.785	1.378	0.935	3.118	0.818	1.445	0.985	3.248	0.02620	0.09830	0.00610	0.13060
V	0.712	1.210	0.833	2.775	0.695	1.190	0.825	2.710	0.04170	0.11600	0.00890	0.16660
W	0.440	0.754	0.521	1.715	0.437	0.752	0.519	1.708	0.01700	0.05060	0.00560	0.07320
X	0.483	0.833	0.603	1.941	0.487	0.853	0.588	1.928	0.01080	0.03930	0.00430	0.05440

Cu, Cr and As ratios did not change during the 24 treatments. This shows that although hot CCA solution was used for all trials, solution disproportionation did not occur. Comparison of treating solution elemental ratios with initial CCA oxide concentration elemental ratios shows that the elemental ratios remained substantially constant (see Table 5 10).

Table 10

Treatment No	Solution Before Treatment Cu	Solution Before Treatment Cr	Solution Before Treatment As
	Ratio%	Ratio%	Ratio%
CCA Oxide Concentrate	25.1	44.0	30.9
A	25.7	43.4	30.9
B	25.0	44.4	30.6
C	25.7	44.6	29.8
D	25.2	45.6	29.3
E	25.6	44.8	29.6
F	25.7	44.6	29.7
G	24.2	45.2	30.6
H	25.9	45.3	28.8
I	25.2	43.8	31.0
J	26.0	44.7	29.3
K	25.7	45.4	28.9
L	25.5	44.5	30.0
M	26.0	44.2	29.8
N	24.9	46.0	29.1
O	25.0	47.3	27.8
P	25.4	45.8	28.8
Q	24.8	44.9	30.4
R	25.0	44.0	31.0
S	25.2	44.1	30.7
T	25.4	44.8	29.9
U	25.2	44.2	30.6
V	25.7	43.6	30.7
W	25.7	44.0	30.4
X	24.9	44.0	31.1

The invention can successfully treat air- and kiln-dried, gauged and ungauged, steamed, square sawn, round and half round radiata pine timber when performed as a combined impregnation and fixation process. Treatments achieved comply to standard MP 3640 and Hazard Classes H3, H4, H5 and H6.

5

The same working solution (with 'top-ups') was used for all 24 treatments and remained in very good condition with no apparent sludging. The only sludge which has appeared on the timber has been a function of resin bleed from the timber not from the working solution.

10

The above Examples are intended to illustrate use of the invention. The invention may be practised with numerous modifications and variations as will be apparent to those skilled in the art. For example numerous types of wood samples, treatment times, chemicals, concentrations of chemicals and treatment times may be used.

CLAIMS

1. A method of impregnating wood or wood based products with a preservative or other wood modifying chemical under pressure comprising:
 - a) impregnating a composition comprising said chemical into the wood under 5 hydraulic pressure and controlling the hydraulic pressure to control the amount of chemical impregnated into the wood;
 - b) replacing the hydraulic pressure with sufficient pneumatic pressure to hold the impregnated composition in the wood while the unused composition is removed from the treatment vessel; and
- 10 c) increasing the pneumatic pressure to force the chemical further into the wood.
2. A method according to claim 1 including finally subjecting the wood to a vacuum sufficient to remove any residual liquid from the surface of the wood to clean said 15 surface.
3. A method according to claim 1 or claim 2, including heating the composition prior to impregnation into the wood.
- 20 4. A method according to claim 3, wherein the composition is heated to a temperature in the range 60°C to 95°C prior to impregnation into the wood.
5. A method according to any preceding claim wherein a pressure in the range 20 to 2000 kPa is used in the hydraulic pressure phase.

6. A method according to claim 5 wherein a pressure in the range 400 to 2000 kPa is used in the hydraulic pressure phase.

7. A method according to claim 6 wherein a pressure in the range 600 to 1500 5 kPa is used in the hydraulic pressure phase.

8. A method according to any one of claims 5-7 wherein the pneumatic pressure used while the unused composition is removed from the treatment vessel is at least as high as the hydraulic pressure which it replaces.

10

9. A method according to claim 8 wherein the pressure of the pneumatic phase while the unused composition is removed from the treatment vessel in the range 20 to 2000 kPa.

15 10. A method according to claim 9 wherein said pneumatic pressure while the unused composition is removed from the treatment vessel in the range 400 to 2000 kPa.

11. A method according to claim 10 wherein the pneumatic pressure while the unused composition is removed is in the range 600 to 1500 kPa.

20 12. A method according to any one of claims 8-11 wherein after removal of the unused composition, the pneumatic pressure used is at least 30% more than the pressure used during the hydraulic pressure phase.

25

13. A method according to claim 12 wherein after removal of the unused composition, the pneumatic pressure used is at least 80% more than the pressure used during the hydraulic pressure phase.

5 14. A method according to any preceding claim wherein the duration of the hydraulic phase is in the range 5 to 240 minutes.

15. A method according to claim 14 wherein the duration of the hydraulic phase is in the range 7 to 180 minutes.

10

16. A method according to any preceding claim wherein the duration of the pneumatic phase is in the range 5 to 240 minutes.

17. A method according to claim 15 wherein the duration of the pneumatic phase
15 is in the range 7 to 180 minutes.

18. A method according to any preceding claim wherein the hydraulic pressure used is in the range of 20 to 2000 kPa and after the unused composition is removed from the treatment vessel the pneumatic pressure used is 100-1000 kPa higher than the pressure
20 used during the hydraulic pressure phase.

19. A method according to any preceding claim wherein the pneumatic pressure used to force the chemical further into the wood is
25 (a) at least 30% higher than the pressure used in the hydraulic phase; and
(b) in the range 26 to 2000 kPa.

20. A method according to any preceding claim wherein the pneumatic pressure used to force the chemical further into the wood is

- (a) at least 80% higher than the pressure used in the hydraulic phase and
- (b) in the range 36 to 2000 kPa.

5

21. A method according to claim 19 wherein said pneumatic pressure is used to force the chemical further into the wood is in the range 520 to 2000 kPa.

22. A method according to claim 21 wherein said pneumatic pressure used to force the chemical further into the wood is in the range 780 to 1500 kPa.

23. A method according to any preceding claim wherein the chemical is deposited in the core of the wood at a concentration of at least 75% of that in the outer one third of the cross-section of the wood.

15

24. A method according to claim 23 wherein the chemical deposited in the core of the wood is at a concentration of at least 100% of that of the outer one third of the cross-section of the wood.

20 25. A method according to any preceding claim wherein the time the wood is under hydraulic pressure plus the time the wood is under pneumatic pressure is less than five hours.

26. A method of claim 25 wherein said time is less than two hours.

25

27. A method according to any preceding claim wherein the material to be impregnated is a mixed charge of sapwood and heartwood.

28. A method of any preceding claim wherein said chemical is selected from 5 preservatives, fire-retardants, water repellents, dimensional stabilising agents, and wood hardening agents.

29. A method according to claim 28 wherein said chemical is a chromium based wood preservative.

10

30. A method according to claim 29 wherein the wood preservative is a copper chrome arsenic, copper chrome boron, copper chrome zinc fluoride or copper chrome preservative.

15

31. A method according to claim 30 wherein the preservative is a copper chrome arsenic preservative.

32. A method according to claim 28 wherein said chemical is a fire-retardant.

20

33. A method according to claim 32 wherein said fire-retardant is a borate salt.

34. A method according to any preceding claim wherein the gas used to apply the pneumatic pressure is air or nitrogen.

25

35. A method according to any preceding claim wherein the chemical can become fixed in the wood and wherein the increased pneumatic pressure used to force the chemical further into the wood is maintained for a period sufficient to allow fixation of the chemical in the wood.

5

36. A method of impregnating wood with a chromium based wood preservative comprising:

10 a) heating the preservative solution and impregnating said solution into the wood at a solution temperature in the range 60 to 95°C, under hydraulic pressure;

b) replacing the hydraulic pressure with sufficient pneumatic pressure using air or nitrogen to hold the impregnated preservative in the wood while the unused solution is removed from the treatment vessel to prevent kickback contamination of the unused solution; and

15 c) maintaining the pneumatic pressure at a level at least as high as the pressure in the hydraulic pressure phase

wherein the temperature at which the preservative is impregnated into the wood and the level and time of the hydraulic and pneumatic pressure phases is manipulated to control the 20 amount of preservative deposited in the wood.

37. A method according to claim 36 wherein following removal of the unused chemical, the pneumatic pressure used is higher than the pressure in the hydraulic pressure phase so as to drive the preservative further into the wood.

25

38. A method according to any preceding claim in which said unused composition is collected for subsequent use.

39. A method of impregnating wood with a chromium based wood preservative 5 comprising:

(a) heating the preservative solution and impregnating said solution into the wood at a solution temperature in the range of 60 to 95°C under hydraulic pressure;

(b) allowing a predetermined amount of said solution to be impregnated into the wood;

10 (c) replacing the hydraulic pressure with sufficient pneumatic pressure, using air or nitrogen, to hold the impregnated preservative in the wood while the unused solution is removed from the treatment vessel to prevent kickback contamination of the unused solution;

(d) increasing the pneumatic pressure to force the preservative further into the 15 wood, and

(e) maintaining the pneumatic pressure at a level higher than used in the hydraulic pressure phase for a time sufficient to allow fixation of most of the preservative within the wood.

20 40. A method according to any one of claims 3, 4 and 36-40 wherein prior to application of hydraulic pressure the composition comprising the preservative or other wood modifying chemical is heated and held in contact with the wood for about 5-20 minutes.

41. A method according to any preceding claim wherein following commencement of the pneumatic pressure phase, the pressure vessel is sealed to maintain the elevated pressure without using a compressor.

5 42. Wood or wood based products treated by a method of any preceding claim.

1/2

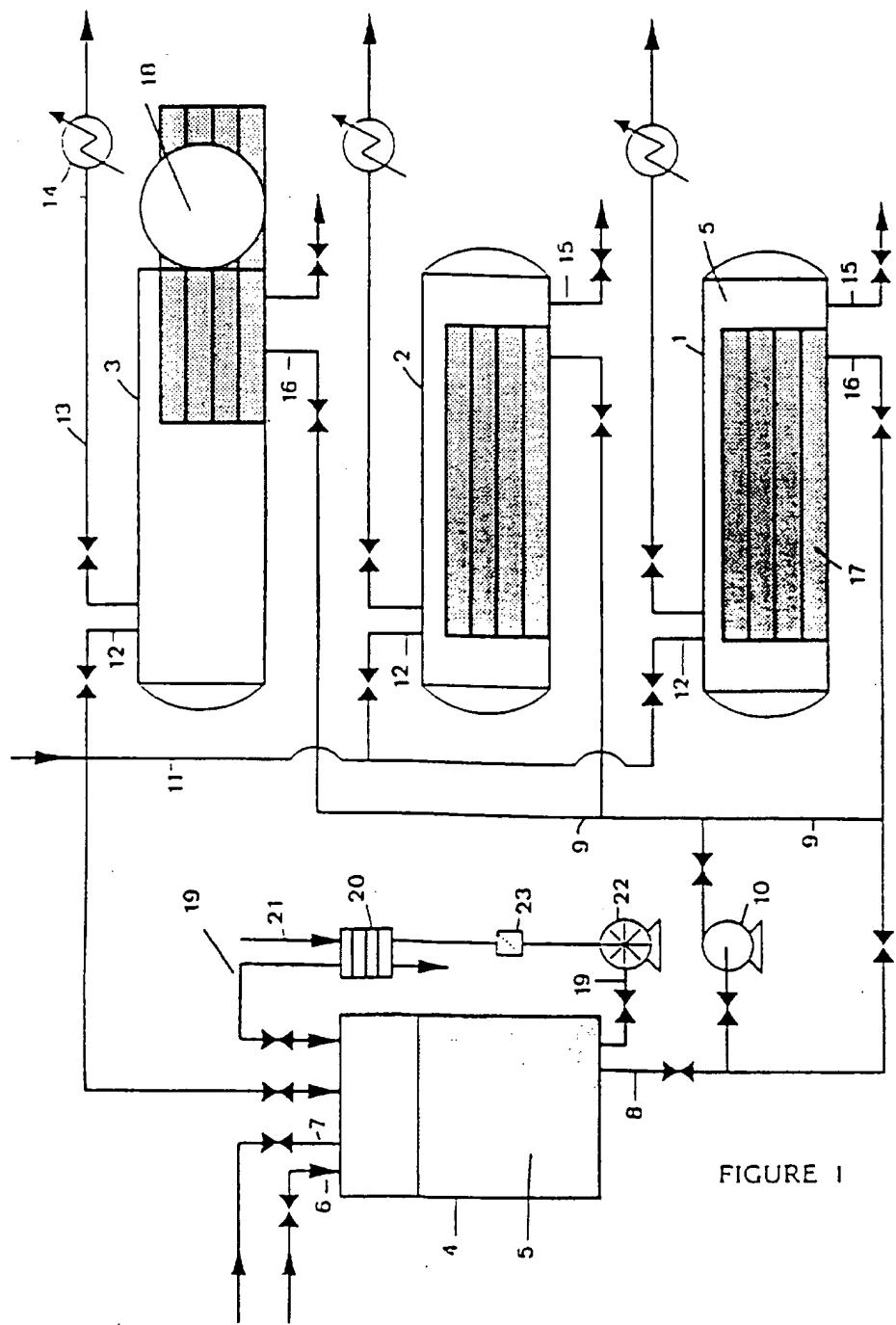


FIGURE 1

2/2

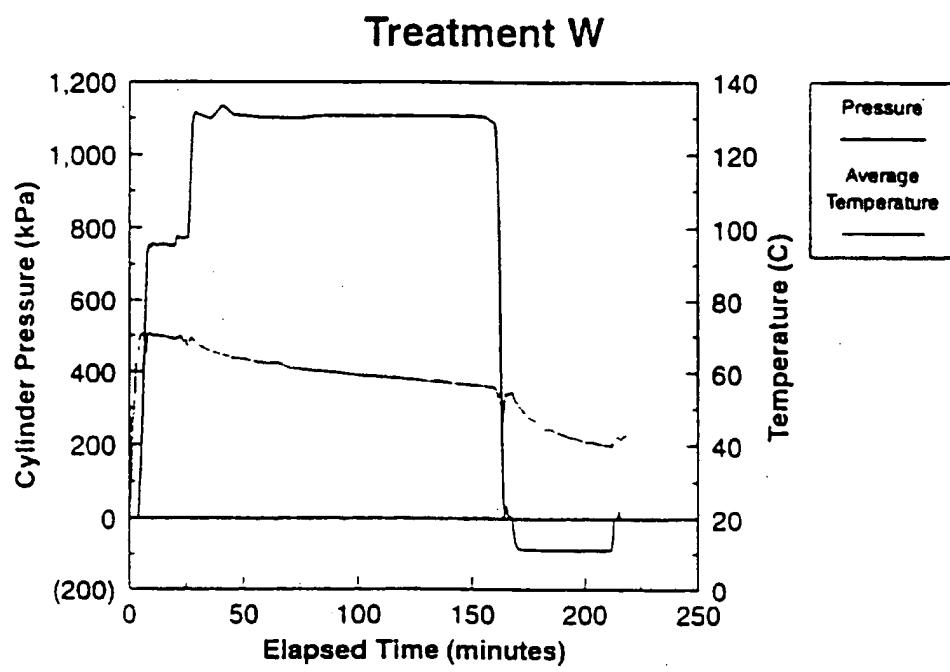


FIGURE 2